

# Deposition Lab / Optics Fabrication

Nathalie Bouet

Beamline Engineering Meeting – March 26<sup>th</sup>, 2019

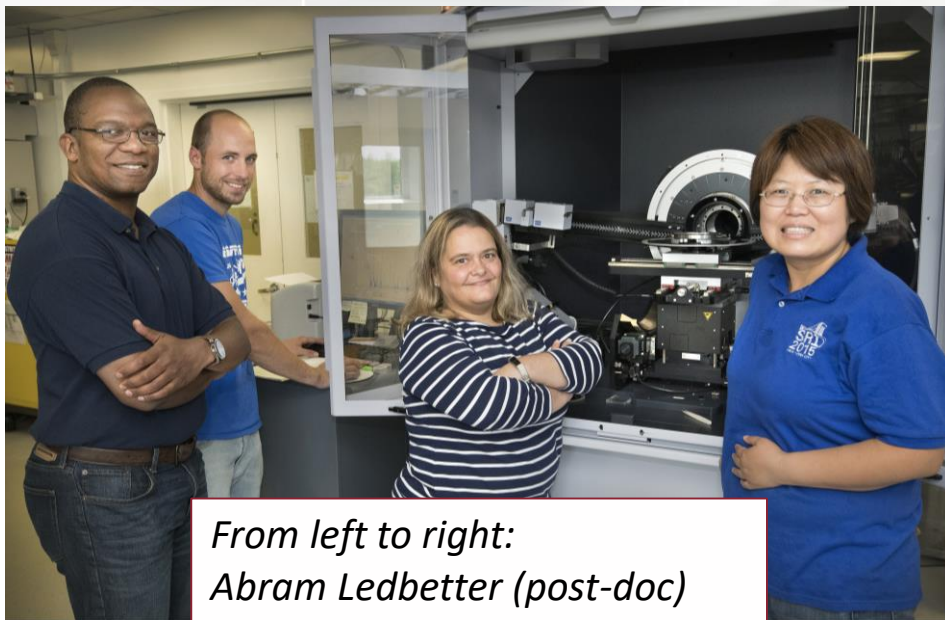


U.S. DEPARTMENT OF  
**ENERGY**

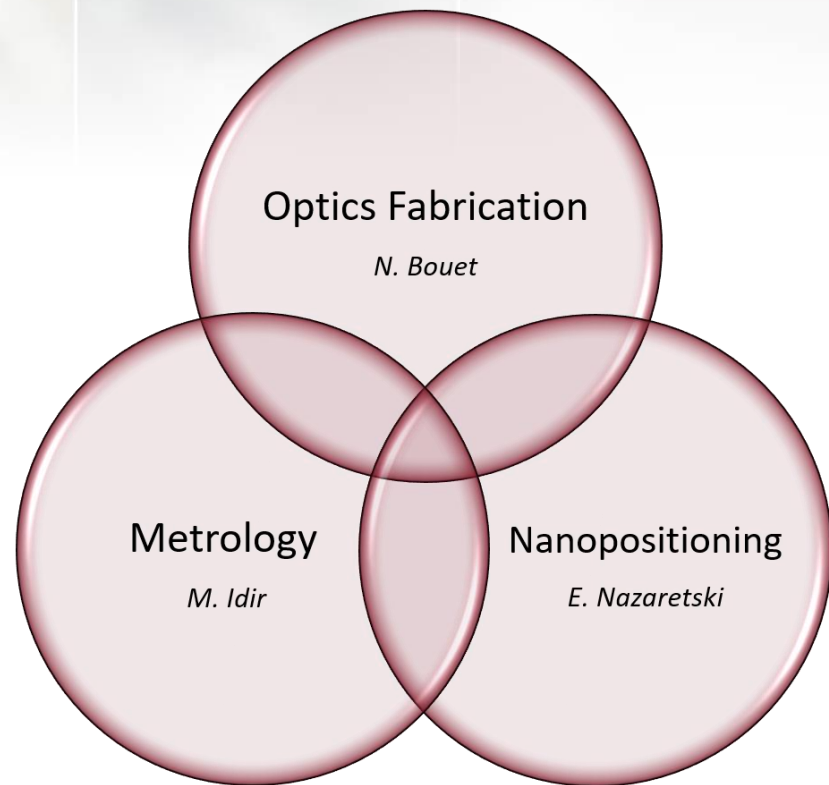
Office of  
Science

**BROOKHAVEN**  
NATIONAL LABORATORY

# Optics Fabrication group



*From left to right:  
Abram Ledbetter (post-doc)  
Matthew Vescovi (technician)  
Nathalie Bouet (scientist)  
Juan Zhou (scientific associate)*



**2010 BNL Engineering Award**

**2015 R&D 100 award** in collab with LBNL, ANL and Abeam Technologies

**2016 R&D 100 award** in collab with NSLS-II nanopositioning team and HXN beamline

**2016 Microscopy Today innovation award** in collab with NSLS-II nanopositioning team and HXN

**US patents 9,153,453 and 9,875,821**

# Collaborators

## Optics and Metrology group

Juan Zhou  
Matthew Vescovi  
Abram Ledbetter  
Evgeni Nazaretski  
Weihe Xu  
Wei Xu  
Dennis Kuhne  
Mourad Idir  
Lei Huang  
Tianyi Wang

## HXN beamline – MLL R&D

Yong Chu  
Hanfei Yan  
Xiaojing Huang

## **Many NSLS-II scientists ...**

Elio Vescovo  
Kon Kaznatcheev  
Claudio Mazzoli  
Eric Dooryhee  
Sanjit Ghose  
Ignace Jarrige  
Valentina Bisogni  
Joe Dvorak  
Pete Siddons  
Lutz Wiegart  
Andrei Fluerașu  
Oleg Chubar...

## **APS-ANL**

Raymond Conley  
Albert Macrander  
Jorg Maser  
Michael Wojcik  
Deming Shu

## **ALS- LBNL**

Valeriy V. Yashchuk

## **Fraunhofer IWS Dresden**

Adam Kubec

## **Accustrata, Inc.**

George Atanasoff  
Christopher Metting  
Hasso Von Bredow

# Optics fabrication at NSLS-II

- Using either additive or subtractive processes

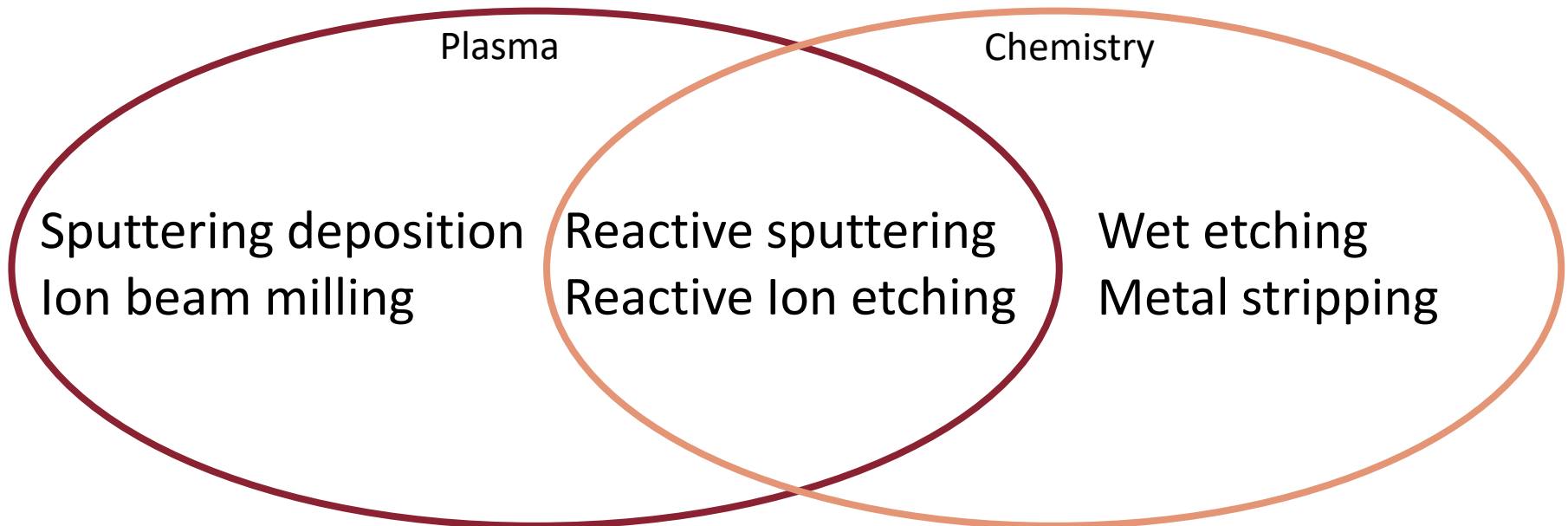


Material deposition



Material removal

- Expertise in plasma and chemistry based techniques

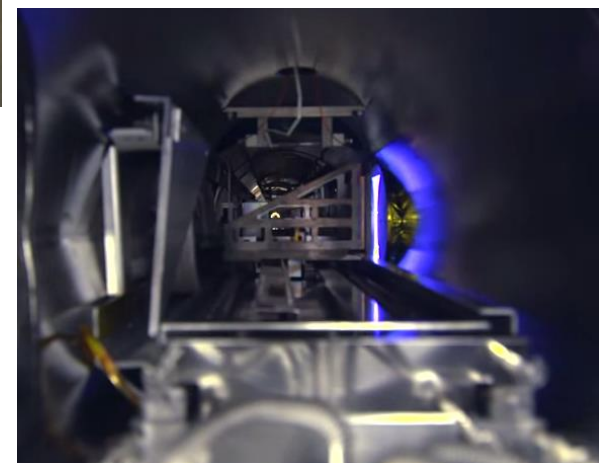
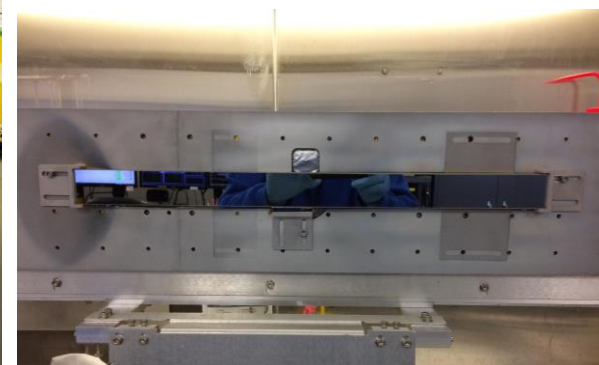




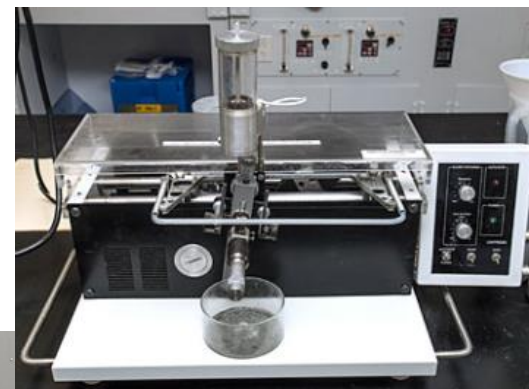
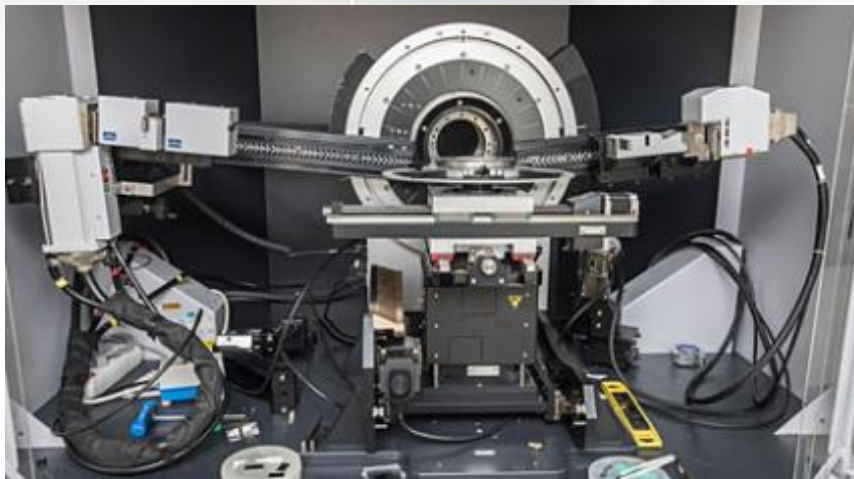
# Deposition Lab



**9 DC Sputtering guns**  
**Linear travel**  
**Sequential cathodes activation**  
**Reactive sputtering possible**  
**Ion gun – capable of handling gas mixtures**  
**Base pressure  $\sim 8 \times 10^{-8}$  Torr**



# Characterization tools and etching lab



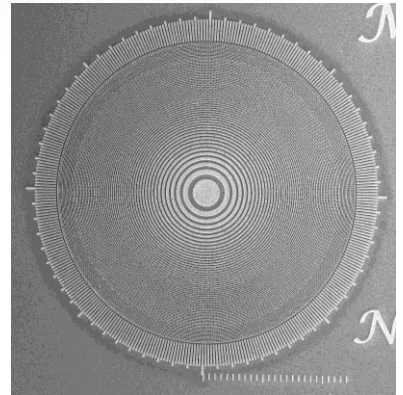


# Multilayer Laue lenses R&D

## High resolution nanofocusing optics for hard XR

MLL propose a solution to **overcome the aspect ratio challenge of ZP for hard X-ray** by creating the zone structure

Fabricated via **deposition of depth graded multilayer** obeying the ZP law and further sectioning to the desired section depth



- **Zone structure created via deposition of depth graded multilayer obeying the ZP law**
  - several thousands of layers to be deposited
  - total film thickness of tens of microns
- Based on thin film deposition techniques capabilities
  - **1nm zone/layer thickness feasible**
- Virtually **no limit on aspect ratio** (thinnest zone width versus section depth ) → Usable for hard X-ray nanofocusing

# Multilayer Laue lenses (MLLs)

Full optics fabrication, metrology & testing in house

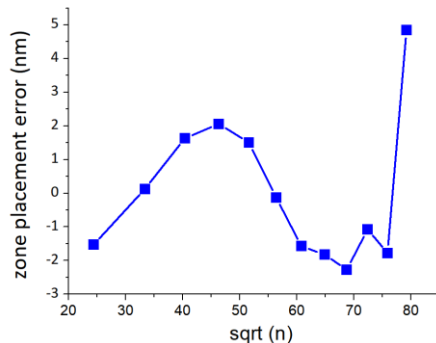
## Deposition



Thousands of layers  $\sim$  5000-8000  
ML up to 100 $\mu$ m thick

## Flat & Wedged geometries

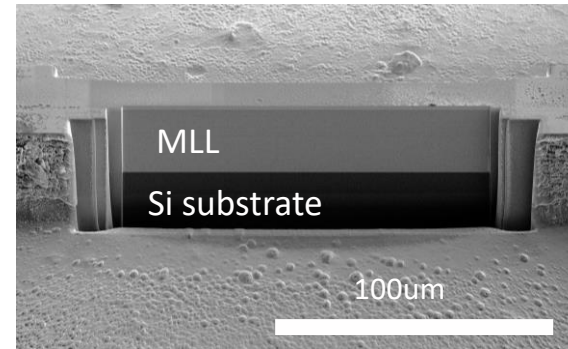
Accuracy of  $\pm$  5nm over 43 $\mu$ m



## Sectioning

via Manual polishing + FIB or RIE + FIB

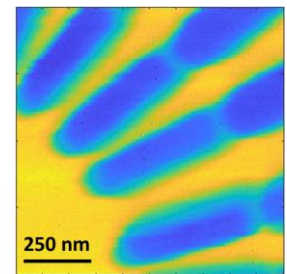
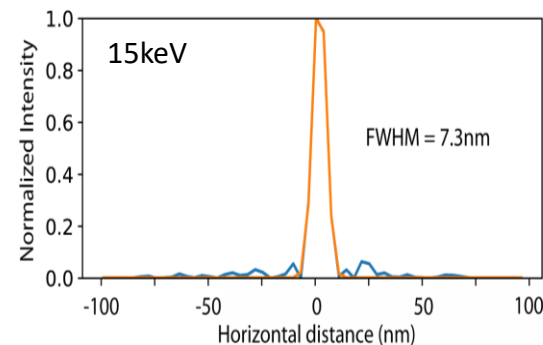
Section depth typically 5 to 15 $\mu$ m for 10-20keV



US patent 9,153,453

## Optics testing

at HXN beamline

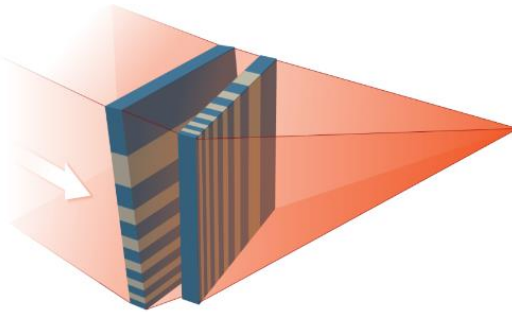


STXM

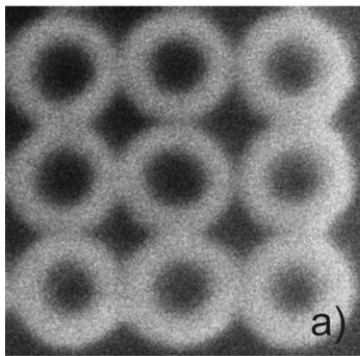


# Optics integrated at Hard Xray Nanoprobe (HXN) beamline for users experiments

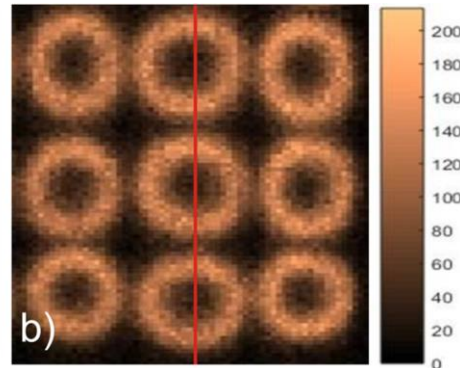
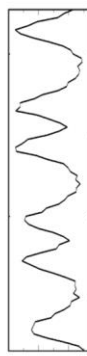
2D focusing achieved  
by using  
2 crossed MLL



Pt circles are 80 nm diameter and 20 nm line width



SEM



XRF (Pt L-edge)  
12keV  
Exposure time 0.2s  
5nm per pixel



*Nazaretski et al., AIP Conf. Proc. 1764 (2016)*

**13 x 13 nm<sup>2</sup> point focus offered to  
NSLS-II users at HXN using a 43um  
and a 53um MLLs**

# Beamline support - Multilayers and thin films coatings



multilayers



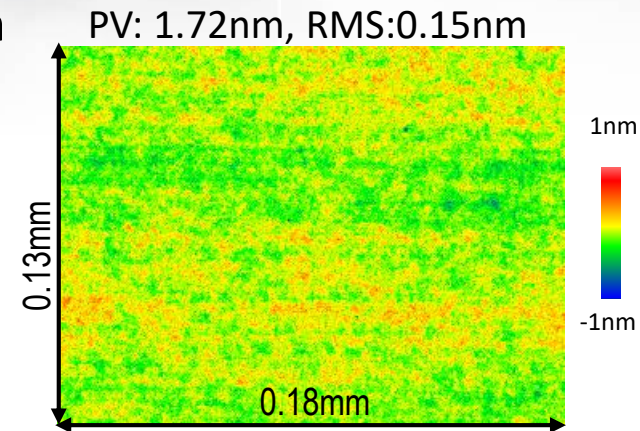
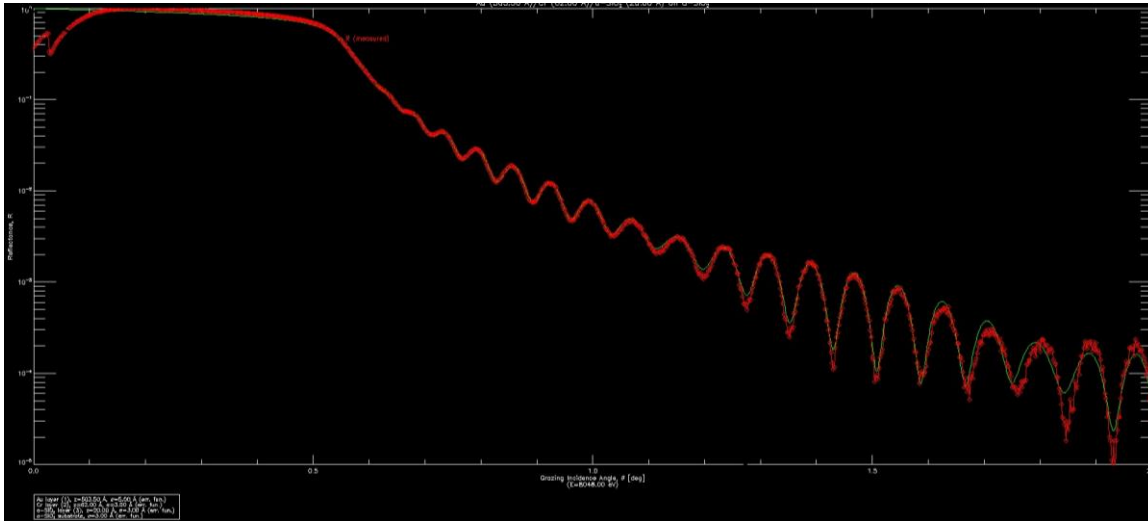
thin films



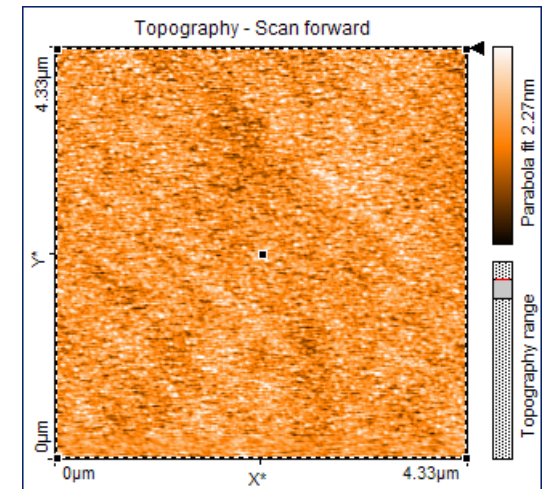
multi-stripes

## Example : gold coatings

Coatings of different substrates from 10mm to 100mm  
Thicknesses from few nm to 1 micron  
for diverse applications



after sys error and best plane subtraction,  
; ZYGO NewView x20 objective, x2 zoom  
lens



AFM roughness (rms) = 0.3nm

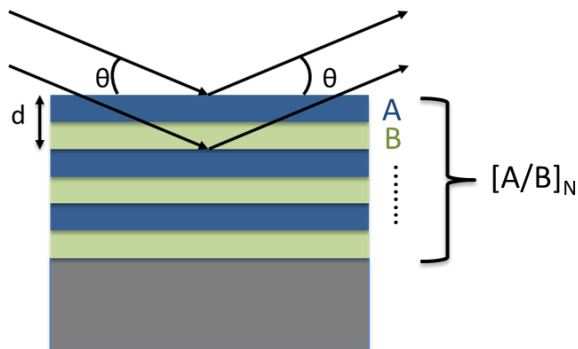
National Synchrotron Light Source II ■



# Multilayers coatings

Example of multilayers grown for NSLS-II ESM and SIX beamlines

Multilayer structure



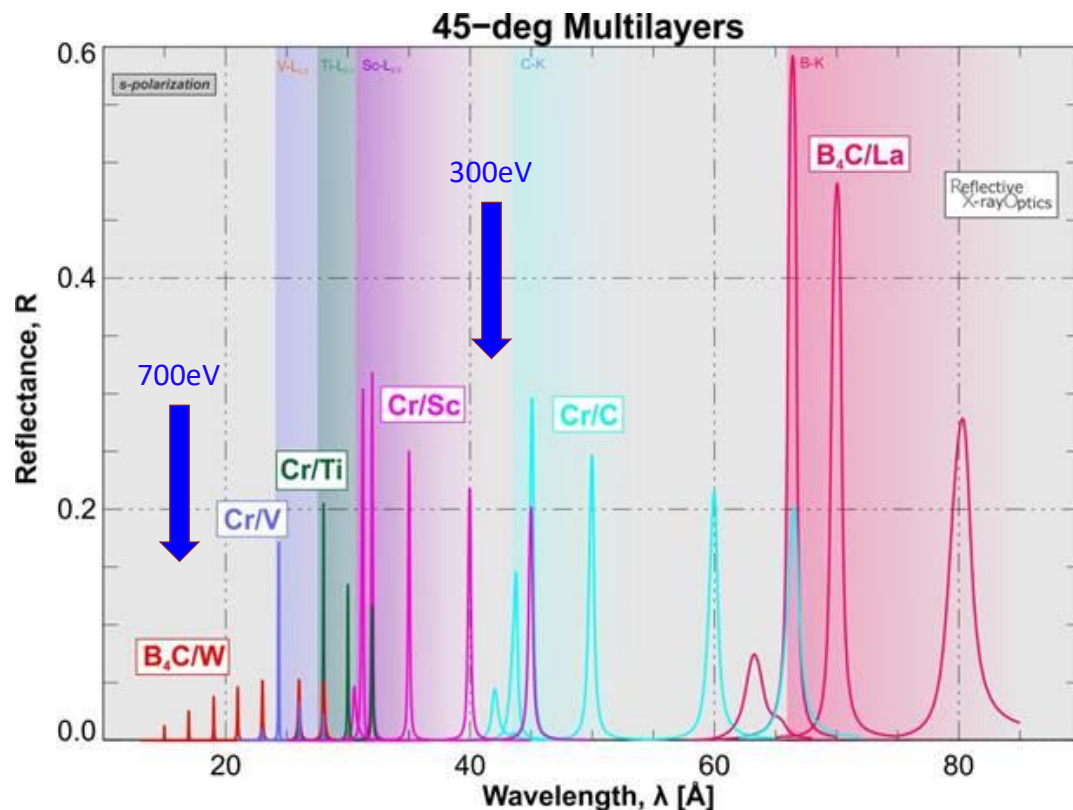
Bragg peaks:  $m \lambda = 2 d \sqrt{n^2 - \cos^2 \theta}$

Bandwidth:  $\Delta E/E \sim 1/N$

Multilayers used as monochromators to characterize their undulators and help with the alignment of beamline elements.

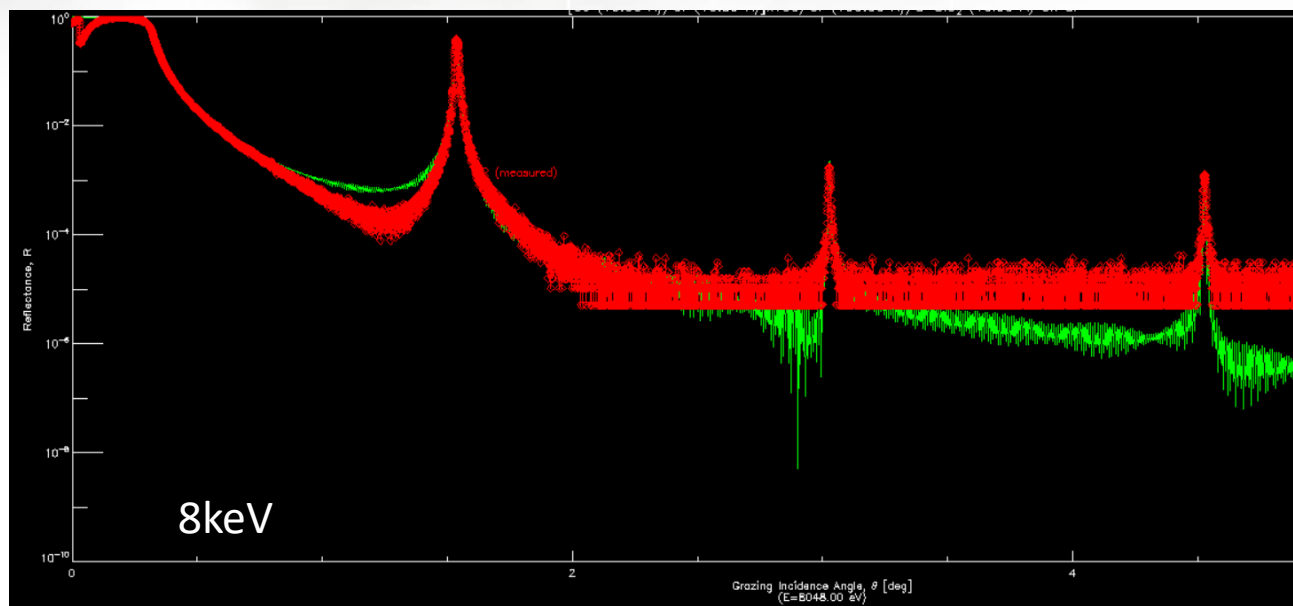
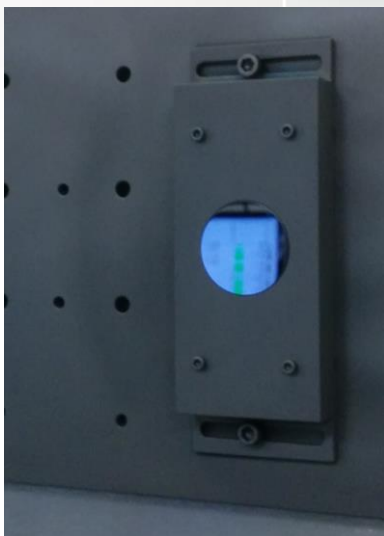
DiagOn reference :

K. Desjardins, AIP Conf. Proc. **879**, 1101 (2007)

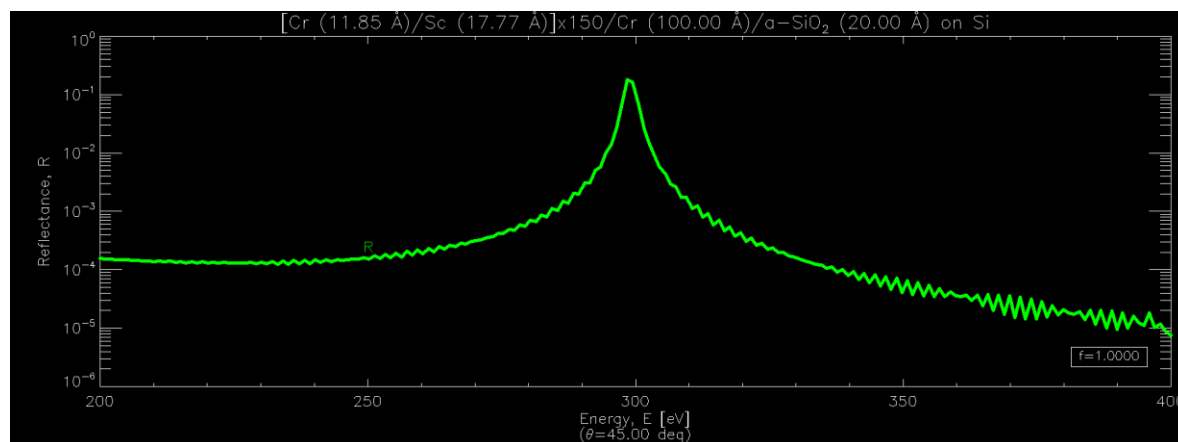


Source : <http://www.rxollc.com>

# 300eV multilayers – 45° incidence



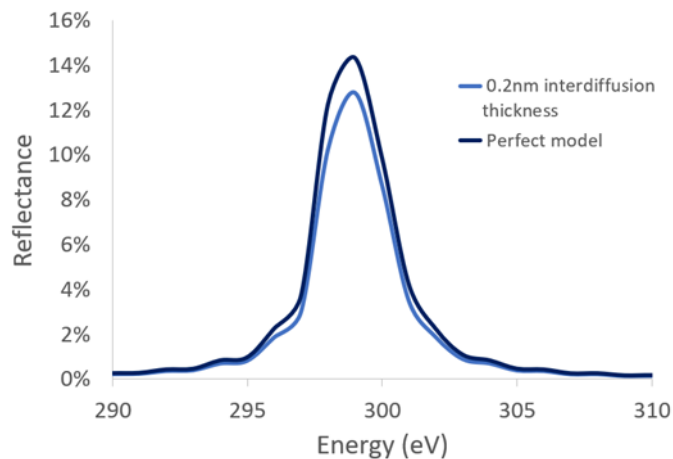
Cr/Sc  
150 bilayers,  $d=2.96\text{nm}$   
Si substrate



# Optics testing of the multilayers @ 45° incidence

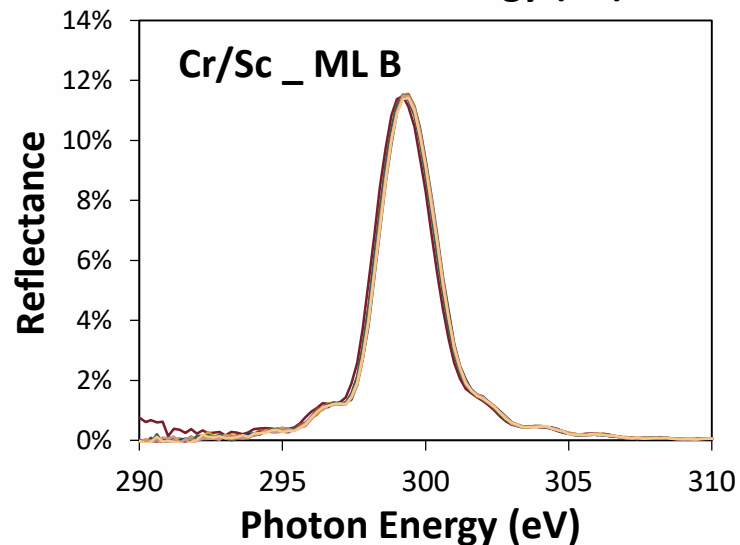
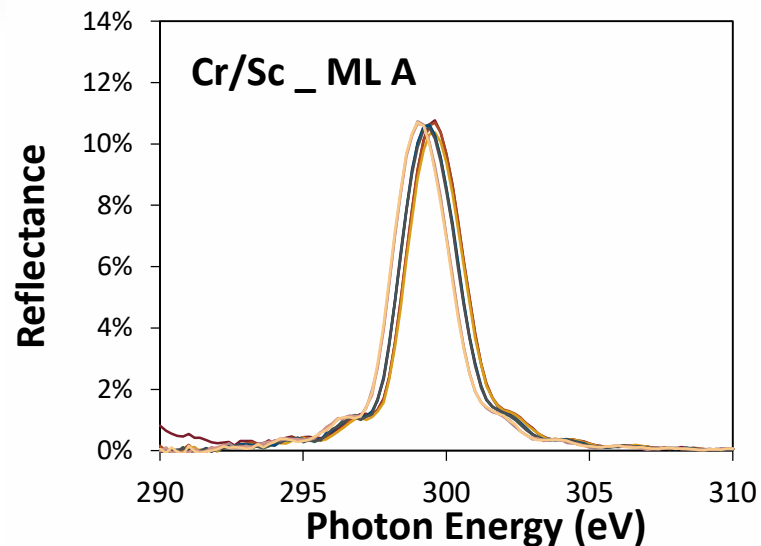


Cr/Sc  
150 bilayers  
 $d=2.96\text{nm}$   
Si substrate



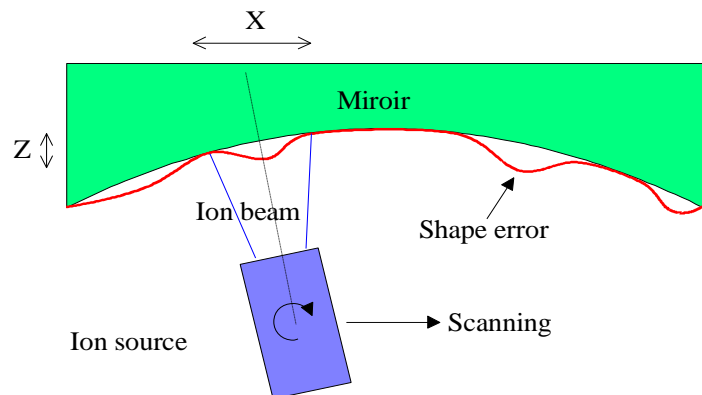
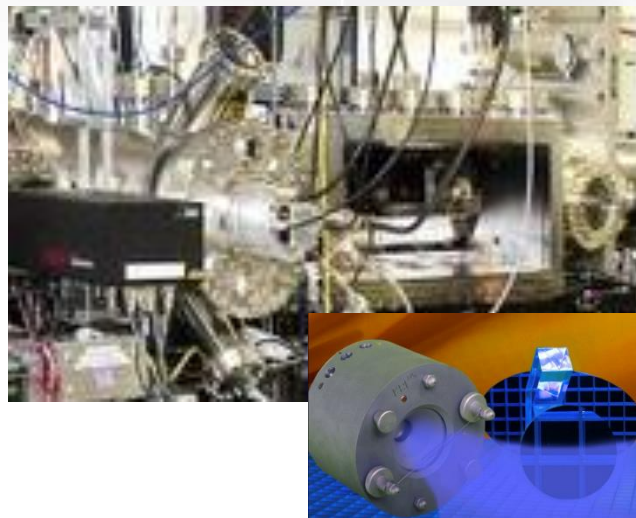
Simulations using CXRO website

MLs tested at ALS (BL 6.3.2)  
at 45 deg incidence by E. Gullikson



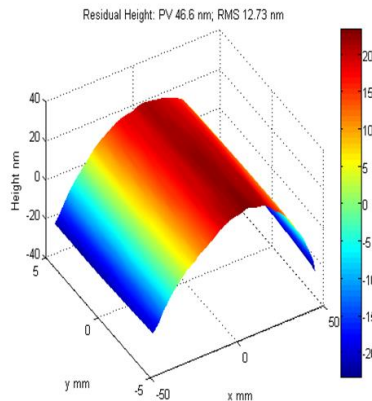


# Ion beam figuring – collab with Metrology



Plane sample #1

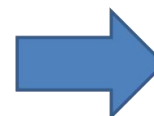
## Promising proof-of-principle first results



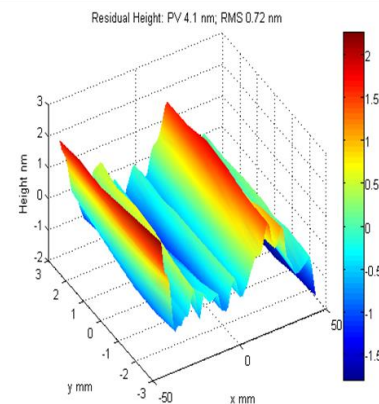
$\lambda/14 \sim 300\$$

$\sim 44 \text{ nm PV}$   
 $\text{RMS: } 15 \text{ nm}$

1D IBF

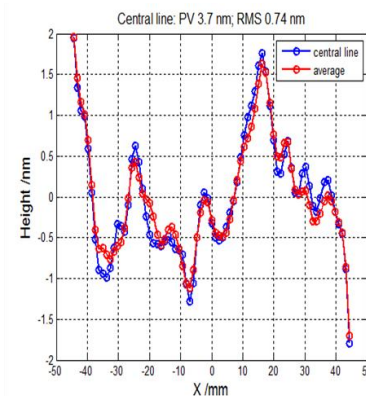
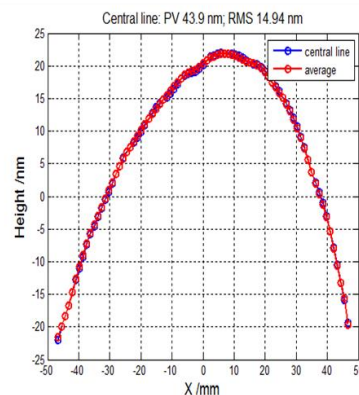


9 minutes



$\sim 3.7 \text{ nm PV}$   
 $\text{RMS: } 0.74 \text{ nm}$

$\lambda/171 \sim 3000\$$



*Review of Scientific Instruments* 86, 105120 (2015)

*J. Synchrotron Rad.* 23, 182 (2016)

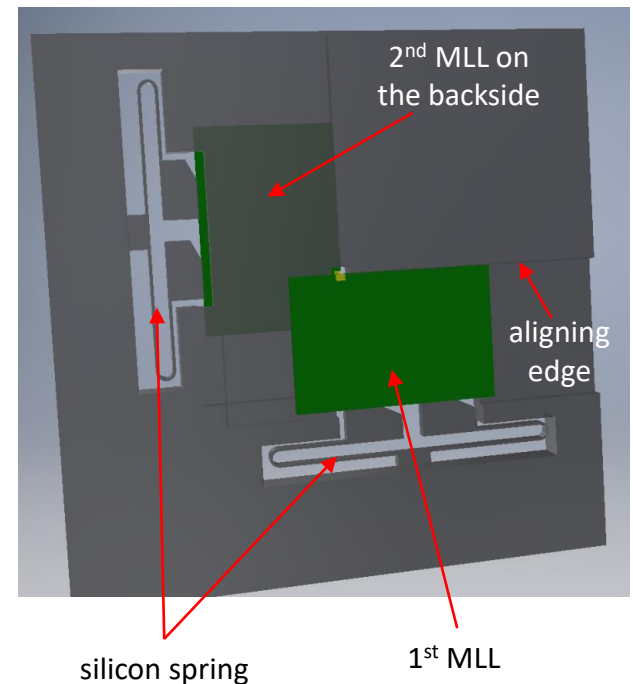
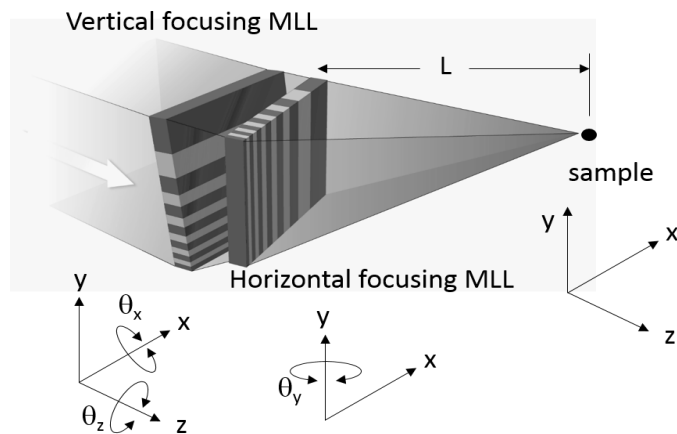
*J. Synchrotron Rad.* 23, 1087 (2016)

New dedicated chamber under procurement

# Bonded MLLs – collab with Nanopositioning

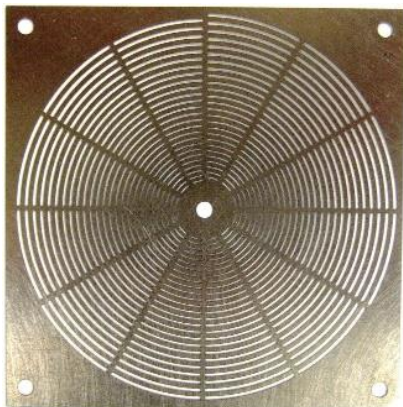
## Monolithic MLL as an alternative to complex alignment procedure

- Alignment of two linear MLL optics is complex
- Stringent requirements for accuracy of alignment and stability of the microscope

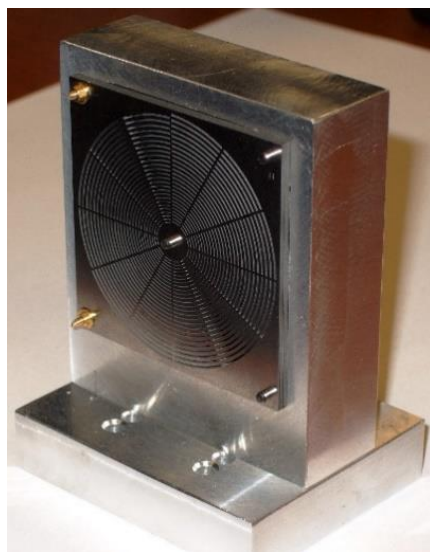


# Conical Slit fabrication for Probing Buried Volumes for Dynamic Measurements of Heterogeneous Materials – collab with XPD beamline

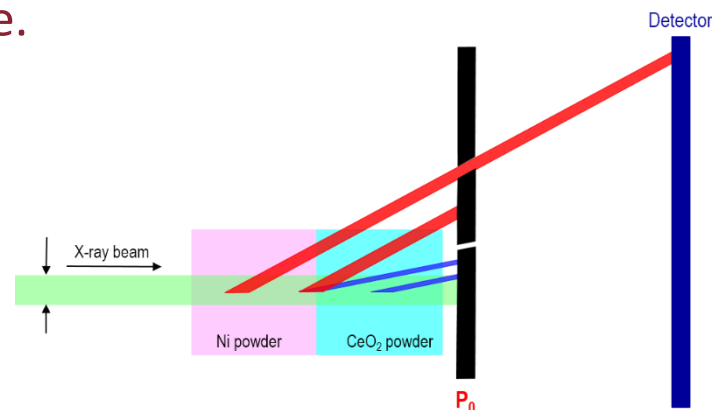
**Goal** : Implementation of a conical/spiral slit which will allow diffracted rays to be transmitted if and only if they originate from a three dimensional gauge volume, defined by the beam size and slit opening size.



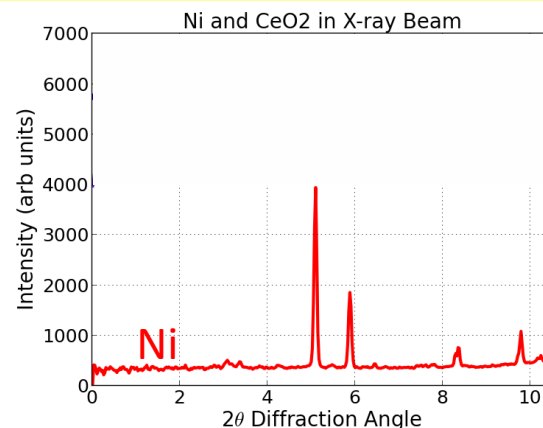
US patent 9,875,821



Fabrication using laser cutting and reactive ion etching → Fabrication methods allowing high accuracy



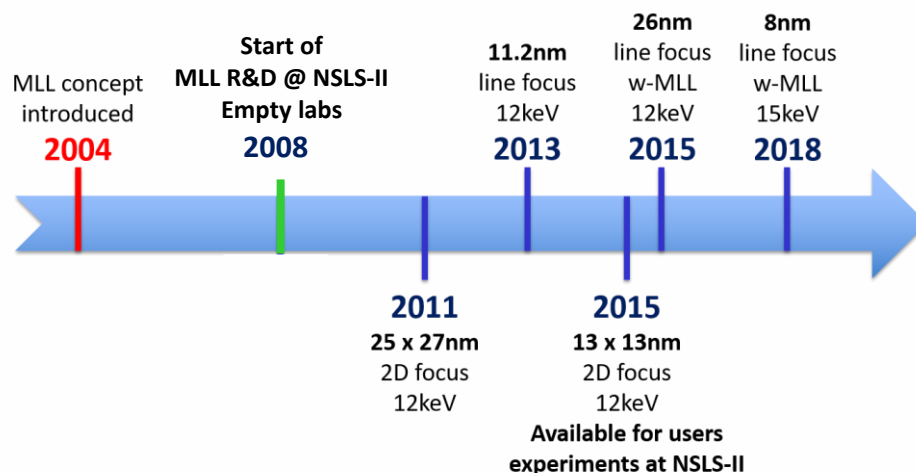
Measure of integrated intensity as the slit gauge volume is translated from a Ni powder to a CeO<sub>2</sub> powder





# Summary

- Capabilities of deposition of high quality films at NSLS-II
  - Simple coatings (Au, Pt, Rh...)
  - Multilayers
  - MLLs
- Very successful R&D on MLL optics with a lot of developments in the past years leading to :
  - Growth of high quality multilayers
  - Demonstration that large aperture MLLs optics are possible
  - High spatial resolution and high efficiency can be achieved with wedged MLLs



# Summary

- Application of our knowledge on fabrication processes to other optical components and new techniques development
  - Bonded MLLs
  - Ion Beam figuring
  - Spider web slits
  - Growth monitoring instrumentation developments ...